भारतीय मानक
IS 12540 : 2023
Indian Standard

ऐक्रायलोनाइट्राइल — विशिष्टि

(पहला पुनरीक्षण)

Acrylonitrile — Specification

(First Revision)

ICS 71.080.30

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Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian (First Revision) Standard was adopted by the Bureau of Indian Standards after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Acrylonitrile ($CH_2 = CH\text{-}C{\equiv}N$) is a highly toxic mobile colourless liquid. It is also known as propenenitrile and vinyl cyanide. It is used in the manufacture of synthetic rubber, plastics, synthetic fibres, etc. Acrylonitrile also finds wide applications in organic synthesis and grain fumigants.

This standard was published in 1988, by deriving assistance from GOST 11097:1964 'Acrylonitrile, commercial grade', issued by the Committee on Standards, Measures and Measuring Instruments of the USSR.

In this (*first*) revision, the test method for determination of colour, refractive index and acetonitrile have been modified. Alternate methods for determination of assay (by GC), water content, hydrocyanic acid content, acetaldehyde content, monoethylether of hydroquinine, iron content and heavy metals have been incorporated. Characteristics of impurities present in acrylonitrile including acetone, benzene, crotononitrile, methyl acrylonitrile, propionitrile and acrolein have been incorporated and characteristic of ammonia content has been deleted.

Clause **3.2.1** of this standard calls for an agreement between the purchaser and the supplier regarding the use of an alternate stabilizer.

Acrylonitrile is hazardous to the health and dangerous to handle. It is advisable to use acrylonitrile in a well-ventilated hood. Code of safety for handling this chemical has been separately given in IS 11141:1984.

The composition of the committee, responsible for the formulation of this standard listed in Annex S.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ACRYLONITRILE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and testing for acrylonitrile.

2 REFERENCES

The standards listed in Annex A contain provisions which through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on these standards are encouraged to investigate the possibility of applying the most recent editions of the standards listed in Annex A.

3 REQUIREMENTS

3.1 Description

The material shall be colourless liquid free from suspended matter.

3.2 The material shall also comply with the requirements given in Table 1, when tested as prescribed in co1 (4) and col (5) of the Table 1.

3.2.1 On agreement between the purchaser and the supplier, hydroquinone in acrylonitrile equivalent to 0.1 percent of the mass may be used for stabilizing the product instead of monoethylether of hydroquinone (MEHQ). In this case, the product may have a yellow tint. The amount of hydroquinone added shall be guaranteed by the manufacturer. A method of test for determination of hydroquinone content has been given in Annex Q for guidance purposes.

3.2.2 The acetonitrile content shall not be determined in acrylonitrile obtained from hydrocyanic acid and acetylene.

3.2.3 *Quality of Reagents*

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4 PRECAUTIONS IN STORING AND HANDLING

4.1 As acrylonitrile is flammable, necessary safeguards against the risk arising from storage and handling of large volumes of flammable liquids shall be provided and all due precautions taken at all times to prevent accident by fire or explosion. It shall be stored in a cool place. Open flames and smoking shall not be allowed where it is stored or handled. Storage vessels shall be vented to safe atmosphere.

4.2 Except when they are opened for the purpose of cleaning and rendering them free from acrylonitrile vapours, all empty tanks and other containers shall be securely closed (*see* IS 11141).

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in mild steel cans and drums. For transportation purposes, it shall be filled in tank cars and tank trucks.

5.2 Marking

5.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of manufacturer and his recognized trademark, if any;
c) Month and year of manufacture;
d) Net mass of the material in the container;
e) Lot or batch number; and
f) Any other statutory requirement.

5.2.2 The containers shall be suitably marked with letter '**TOXIC**' and with symbol as given in Fig. 5 of IS 1260 (Part 1). The label shall also bear the following clear and legible instruction:

'HARMFUL VAPOUR, DO NOT INHALE;

FLAMMABLE KEEP IN COOL PLACE'

5.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

6.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed Annex R.

Table 1 Requirements of Acrylonitrile

(*Clauses* 3.2 *and* R-2.2.6)

Sl No.	Characteristic	Requirements	Method of Test, Ref to	
			Annex	Indian Standard
(1)	(2)	(3)	(4)	(5)
i)	Distillation range at 760 mm Hg:		—	IS 5298
	a) Initial boiling point, *Min*	74.5 ºC		
	b) Dry point, *Max*	79.0 ºC		
ii)	Specific gravity, at 27 °C/27 °C	0.798 to 0.802	B	—
iii)	Colour, Pt-Co scale, *Max*	15	—	IS 1448 (Part 178)
iv)	Refractive index[1] at 25 °C	1.3882 to 1.3892	—	IS 1448 (Part 158)
v)	Water content, percent by mass, *Max*	0.8	C	IS 1448 (Part 175)[2] /IS 1448 (Part 182)
vi)	Assay, percent by mass, *Min*	99.0	D/E[2]	—
vii)	Acetone, ppm, *Max*	50	E	—
viii)	Acetonitrile[3], ppm, *Max*	500	E	—
ix)	Benzene, ppm, *Max*	a) For food contact applications: 10 b) For other applications: 50	E	—
x)	Crotononitrile[1], ppm, *Max*	40	E	—
xi)	Methylacrylonitrile, ppm, *Max*	250	E	—
xii)	Propionitrile, ppm, *Max*	50	E	—
xiii)	Acrolein, ppm, *Max*	10	E	—
xiv)	Hydrocyanic acid content, percent by mass, *Max*	0.001	F	—
xv)	Methyl vinyl ketone, percent by mass, *Max*	0.03	G/E[2]	—
xvi)	Acetaldehyde content, percent by mass, *Max*	0.005	H/E[2]	—
xvii)	Monoethyl ether of hydroquinone (MEHQ), ppm, *Max*	45	J	—
xviii)	Peroxide content, ppm, *Max*	0.2	K	—

Table 1 (*Concluded*)

Sl No.	Characteristic	Requirements	Method of Test, Ref to	
			Annex	Indian Standard
(1)	(2)	(3)	(4)	(5)
xix)	Soluble iron content, percent by mass, *Max*	0.00002	L	IS 1448 (Part 172)[2)]
xx)	Heavy metals (*as* Cu), percent by mass, *Max*	0.00002	M	IS 1448 (Part 172)[2)]
xxi)	*p*H (5 percent aqueous solution)	6 to 7.5	N	—
xxii)	Non-volatile matter, percent by mass, *Max*	0.01	P	—

NOTES

1 The following are the optional parameters to be tested:

a) Sl No. (iv) Refractive index at 25 °C

NOTE — Also, if, for any reason, it is not possible to determine the refractive index at the specified temperature, then the value of refractive index may be calculated by applying suitable correction factor or a factor of 0.000 38 per degree Celsius if the factor is not available.

b) Sl No. (x) Crotononitrile

2 In case of disputes, the following methods shall be the referee methods:

a) Annex E for determination of assay, methyl vinyl ketone and acetaldehyde content;

b) IS 1448 (Part 175) for determination of water content; and

c) IS 1448 (Part 172) for determination of heavy metals and soluble iron content.

3 *See* **3.2.2**.

ANNEX A

(*Clause* 2)

LIST OF REFERRED STANDARDS

IS No.	*Title*
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
IS 1448	Methods of test for petroleum and its products:
(Part 158) : 2016/ ISO 5661 : 1983	Part 158 Hydrocarbon liquids — Determination of refractive index
(Part 172) : 2020	Part : 172 ICP-AES Method for determination of trace elements in petroleum products
(Part 175) : 2020 / ISO 6296 : 2000	Part 175 Petroleum products — determination of water — Potentiometric karl fischer titration method
(Part 178) : 2020/ISO 6271 : 2015	Part 178 Clear liquids — Estimation of colour by the platinum cobalt colour scale
(Part 182) : 2020 / ISO 12937 : 2000	Part 182 Petroleum products — determination of water — Coulometric karl fischer titration method
IS 2362 : 1993	Determination of water by karl fischer method — Test method (*second revision*)
IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)
IS 5298 : 2013	Method for determination of distillation range and distillation yield (*second revision*)
IS 11141 : 1984	Code of safety for acrylonitrile

ANNEX B

[*Table* 1 and *Sl No.* (ii)]

DETERMINATION OF SPECIFIC GRAVITY

B-1 APPARATUS

B-1.1 Pyknometer or Specific Gravity Bottle — 25 ml capacity

B-1.2 Water-Bath — maintained at (27.0 ± 0.2) ºC.

B-2 PROCEDURE

Clean and dry the pyknometer or specific gravity bottle. Weigh it, fill it with freshly boiled distilled water, place it in the bath maintained at (27.0 ± 0.2) °C and allow sufficient time (about 45 min) to attain the temperature of the bath. Then insert the capillary stopper which has been brought to (27.0 ± 0.2) °C. Wipe the excess liquid from the stopper, remove the pyknometer or the specific gravity bottle from the bath, bring to room temperature and weigh. Empty the pyknometer or specific gravity bottle, clean and dry it, and repeat the operation with the material at (27.0 ± 0.2) °C.

B-3 CALCULATION

$$\text{Specific gravity at 27 °C/27 °C} = \frac{A-B}{C-B}$$

where

A = mass of the pyknometer or specific gravity bottle filled with the material, in g;

B = mass of the dry pyknometer or specific gravity bottle, in g; and

C = mass of the pyknometer or specific gravity bottle filled with water, in g.

NOTE — Digital density meter may also be used.

ANNEX C

[*Table* 1 and *Sl No.* (v)]

DETERMINATION OF WATER CONTENT

C-1 GENERAL

Three method namely, IS 1448 (Part 175) determination of water content by potentiometric karl fischer titration method; IS 1448 (Part 182) determination of water content by coulometric karl fischer titration method and Method A determination of water content by IS 2362 have been prescribed. In case of disputes, IS 1448 (Part 175) shall be the referee method.

C-2 METHOD A

C-2.1 Apparatus

C-2.1.1 *Pipette* — with a mark at 1.1 ml to 1.5 ml.

C-2.2 Procedure

Take 0.5 g to 1.5 g weighed portion with pipette. Determine the moisture content by Karl Fischer method as given in IS 2362.

ANNEX D

[*Table* 1 and *Sl No.* (vi)]

DETERMINATION OF ACRYLONITRILE CONTENT (ASSAY) BY TITRATION METHOD

D-1 APPARATUS

D-1.1 Conical Flasks (*see* Fig. 1)

D-1.2 Pipette — with a mark at 1.1 ml to 1.5 ml

D-2 REAGENTS

D-2.1 Sodium Sulphite Anhydrous Solution — 2 N

D-2.2 Standard Sulphuric Acid Solution — 1 N

D-2.3 Thymolphthalein Alcoholic Solution — 0.1 percent

D-3 PROCEDURE

Weigh the 250 ml conical empty flask to the nearest 0.0 002 g. Now fill the pipette up to the mark with the product under test. Transfer the content of the pipette to the above weighed conical flask and reweigh the content with the same accuracy. The weighed portion shall be found from the difference between the two weighing. The weighed portion shall not be more than 1.2 g. Now add 50 ml of neutral sodium sulphite solution and 25 ml of water to the flask and quickly close the flask with its stopper and keep it for 3 min in warm water (at 30 to 35 °C), shaking it gently from time to time without removing it from water. After 3 min have elapsed, remove the flask from the water, shake it up for 1 min. Add 6 or 7 drops of the thymolphthalein solution and titrate the solution under test with standard sulphuric acid solution until blue colour disappears.

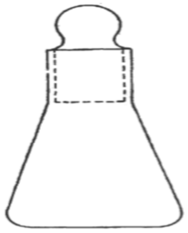

FIG. 1 CONICAL FLASK

D-4 CALCULATION

Acrylonitrile content is calculated as:

$$\text{Acrylonitrile content, percent by mass} = \frac{V \times 0.053 \times 100}{M}$$

where

V = volume of standard sulphuric acid solution (1 N) used in the titration, in ml;

0.053 = amount in g of acrylonitrile corresponding to 1 ml of standard sulphuric acid solution (1 N); and

M = mass of the material taken for the test, in g.

NOTE — A blank test shall be carried out under the test conditions specified above in order to check the sodium sulphite for neutrality. The solution should remain colourless. If the sulphite solution reveals a slightly alkaline reaction, it may be neutralized with a calculated amount of acid and then check for neutrality to thymolphthalein.

ANNEX E

[*Table* 1 and *Sl No.* (vi) to (xiii), (xv) and (xvi)]

DETERMINATION OF ACRYLONITRILE CONTENT (ASSAY) AND ITS IMPURITIES BY GAS CHROMATOGRAPHY METHOD

E-1 APPARATUS

E-1.1 Gas Chromatograph

E-1.1.1 Any gas chromatograph equipped with a flame ionization detector (FID), a split injector and a suitable electronic integrator/software can be used with following accessories and operating condition:

Column	:	Fused silica nonpolar phase; crossbond dimethyl polysiloxane general purpose columns with length 90 m to 120 m; internal diameter 0.53 mm and film thickness 0.3 µm or equivalent.
Carrier gas	:	Helium (purity 99.995 percent, minimum)
Fuel gas and Flow rate	:	Hydrogen (high purity 99.990 percent); Flow rate: 33 ml/min and Air (hydrogen free, high purity); Flow rate : 360 ml/min
Injection volume	:	2 µl
Split flow	:	20 ml/min
Detector	:	FID detector

E-1.1.2 *Temperature Programme of Oven, Detector and Injector:*

Injector Temper-ature, °C	*Detector Temper-ature, °C*	*Ramp Rate, °C/min*	*Oven Temp-erature, °C*	*Hold Time, min*
250	250	—	50	20
		5	100	0
		15	250	5

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard

E-1.2 Analytical Balance — with least count of 0.1 mg

E-1.3 Serum Bottle — with septum, 50 ml

E-1.4 Syringe — 10 µl

E-2 REAGENTS

E-2.1 Isobutanol — 99 percent

E-2.2 Acetone — known purity

E-2.3 Acetonitrile — known purity

E-2.4 Acetaldehyde — known purity

E-2.5 Benzene — known purity

E-2.6 Crotononitrile — known purity

E-2.7 Methacrylonitrile — known purity

E-2.8 Propionitrile — known purity

E-2.9 Methanol — known purity

E-2.10 Methyl Vinyl Ketone — known purity

E-2.11 Acrolein — known purity

E-2.12 Ethanol — known purity

E-3 PREPARATION OF CALIBRATION STANDARDS

E-3.1 Stock Acrylonitrile

Inject 2 µl of high purity acrylonitrile (stock), three times in gas chromatograph (GC). Determine the average area count for each known impurity.

E-3.2 Stock Standard

E-3.2.1 Take 95 ml stock acrylonitrile in 100 ml serum bottle with septum. Weigh to the nearest 0.1 mg and record the initial weight. Use the glass syringe and add the following amount of each component of known purity to the bottle.

Component	*µl*
Acetone	200
Acetonitrile	300
Acetaldehyde	20
Benzene	100
Crotononitrile	600
Methacrylonitrile	600

Component	*µl*
Propionitrile	200
Methanol	200
Methyl Vinyl Ketone	150
Acrolein	10
Ethanol	200

E-3.2.2 Weigh the bottle after each addition and record the weight to the nearest 0.1 mg. After addition of all the components, cap the bottle and shake it to mix the solution. Determine the individual component weight by subtracting the previous weight from the weight after a component is added.

E-3.2.3 *Calculation*

Calculate the added concentration (mg/kg) of each component as:

Component concentration (mg/kg) =

$$\frac{Component\ weight\ (mg) \times component\text{'s}\ known\ purity\ (percent)}{Total\ weight\ (\text{kg})}$$

E-3.3 Internal Standard

E-3.3.1 Take 50 ml water into a 50 ml serum bottle with septum and weigh to the nearest 0.1 mg (W_1). With 1 ml syringe, add 0.25 ml of isobutanol into the bottle. Weigh the bottle after addition and record the weight to the nearest 0.1 mg (W_2). Cap the bottle and shake it to mix the solution.

NOTE — Toluene or any suitable internal standard may also be used as internal standard instead of isobutanol.

E-3.3.2 *Calculation*

Calculate the isobutanol concentration (mg/kg) as:

Isobutanol concentration (mg/kg) =

$$\frac{W_2 - W_1}{W_2} \times \frac{Isobutanol,\ purity\ (percent)}{100} \times 10^6$$

where

W_1 = initial weight of the beaker after addition of water into the bottle, in g; and
W_2 = final weight of beaker after addition of isobutanol into the bottle, in g.

E-4 CALIBRATION

Prepare a standard solution and internal standard as given in **E-3.2** and **E-3.3**, in the expected concentration range. Make sure the GC instrument is adjusted to the conditions stated as above. Inject 1.0 µl of the standard at least three times and obtain the typical chromatogram (*see* Fig. 2). Calculate the calibration correction factor for each component by dividing its concentration by the respective area in its chromatogram. Find the arithmetic mean of the factors calculated for each component. Each factor is calculated to 4 decimal places.

E-5 PROCEDURE

Add internal standard (isobutanol) to the sample in the same manner as the standards. Inject the sample and calculate the amount of each component.

E-6 CALCULATION

E-6.1 Sum the areas of all unknown components.

E-6.2 Multiply the area of each component of the sample by its relative response correction factor as calculated in **E-4**. Obtain the sum of all corrected areas. Normalize each corrected area to 100 percent and report each as mass percent or use suitable software for auto calculation with respect to calibration standard. The assay shall be determined by deducting the sum of percent content of impurities from 100.

NOTE — Use response correction factor = 1, for the unknown components.

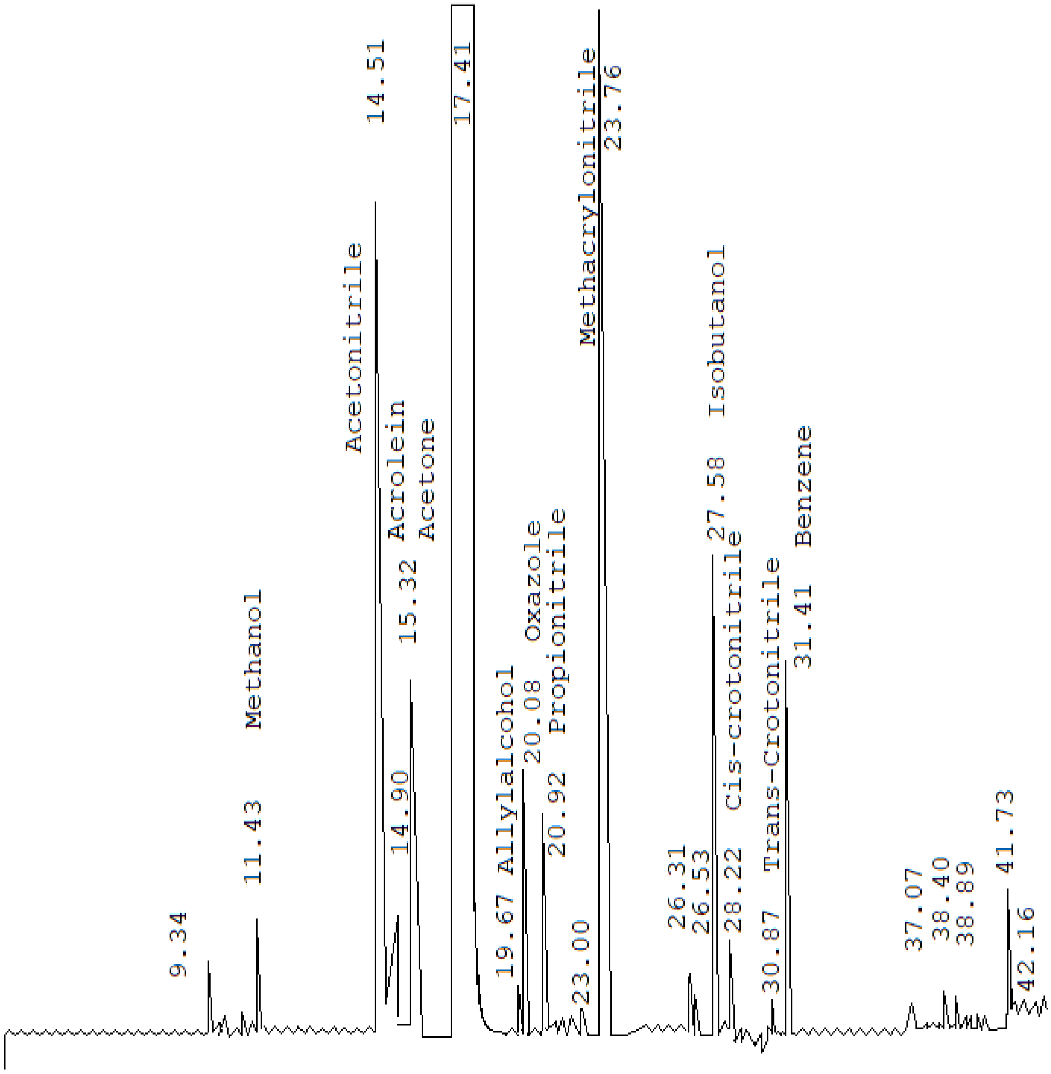

FIG. 2 TYPICAL CHROMATOGRAPH OF ACRYLONITRILE AND ITS IMPURITIES

ANNEX F

[*Table* 1 and *Sl No.* (xiv)]

DETERMINATION OF HYDROCYANIC ACID CONTENT

F-1 GENERAL

Two methods, Method A determination of hydrocyanic acid content by spectrophotometer and Method B determination of hydrocyanic acid content by titration method have been prescribed. In case of disputes, Method A shall be the referee method.

F-2 METHOD A

F-2.1 Apparatus

F-2.1.1 *Spectrophotometer of Photoelectric Colorimeter* — equipped with cells of 5 cm light path

F-2.2 Reagents

F-2.2.1 *Copper Sulphate Solution* — 0.02 percent

F-2.2.2 *Potassium Cyanide*

F-2.2.3 *Potassium Hydroxide Solution* — 0.03 percent

F-2.2.4 *Rectified Spirit* (*see* IS 323)

F-2.2.5 *Buffer Solution*

To prepare the solution, dissolve 2.15 g of disubstituted sodium phosphate in 1 litre of distilled water

F-2.2.6 *Acrylonitrile* — free from hydrocyanic acid

F-2.2.7 *Phenolphthalein Solution*

Dissolve 37.5 g of sodium hydroxide in 750 ml of distilled water and add 25 g of phenolphthalein. Heat the resulting solution to a temperature of 70 °C and, while stirring constantly, add 17.5 g of zinc dust. Heat the solution to boiling and continue the reaction until the solution is completely decolorized. Then cool the solution and add approximately 200 ml of distilled water and then hydrochloric acid until precipitation is complete. Separate the precipitate from the solution by decantation, filter it out on a buchner funnel and wash it with distilled water until the wash water reacts neutral. Then dissolve the precipitate on the filter paper in hot alcohol. After that, add water to the alcoholic solution until a white deposit (phenolphthalein) is completely precipitated. Filter out the precipitate and dry it at a temperature of 110 °C to 120 °C (its melting point is 225 ºC). If necessary, recrystallize. Dissolve the dry phenolphthalein in ethyl alcohol, taking 0.5 g per 100 ml of alcohol.

Introduce 3 ml of the alcoholic phenolphthalein solution into a 100 ml measuring flask and dilute the solution to the mark with the copper sulphate solution. The solution shall be prepared 1 h before use.

F-2.3 Calibration Curve

F-2.3.1 Weigh out 0.6 g of potassium cyanide to the nearest 0.000 3 g, transfer the weighed portion quantitatively to a 1 litre measuring flask, dissolve in water and dilute to the mark. Pipette 5 ml of the resulting solution into a 100 ml measuring flask and dilute with freshly boiled water to the mark (solution A). The content of potassium cyanide in 1 ml of solution A corresponds to a 0.001 2 percent hydrocyanic acid solution which is equivalent to its content calculated for a 3 ml sample of acrylonitrile equal to 0.000 5 percent.

F-2.3.2 To plot the calibration curve, pipette 3.0 ml of acrylonitrile free from hydrocyanic acid into five 50 ml measuring flasks, each containing 20 ml of freshly boiled water and to each flask add 1.0 ml, 2.0 ml, 3.0 ml, 4.0 ml and 5.0 ml of solution A, respectively. Then add to each flask, 10 ml of the buffer solution, 5 ml of the phenolphthalein solution and mix the contents of the flasks thoroughly. Now add 5 ml of the potassium hydroxide solution to each flask, dilute the solution in each flask with water to the mark and mix. Introduce the solution under test into a cell, the height of the liquid being 10 mm, and determine the optical density at a wavelength of 554 nm. Fill a second cell with distilled water for comparison.

F-2.3.3 The values obtained shall be plotted with the optical density as ordinate and the percentage of hydrocyanic acid as abscissa.

F-2.4 Procedure

Introduce 3.0 ml of the material into a 50 ml measuring flask containing 20 ml of freshly boiled water. Add to flask, 10 ml of the buffer solution (**F-2.2.5**), 5 ml of phenolphthalein solution (**F-2.2.7**) and mix the contents of the flask thoroughly. Now add 5 ml of potassium hydroxide solution (**F-2.2.3**) to the flask, dilute the solution with water to the mark and mix. Introduce the solution under test into cell, the height of the liquid being 10 mm and determine the optical density at a wavelength of 554 nm. Fill a second cell with distilled water for comparison.

The solutions shall be tested in the spectrophotometer immediately after they are prepared.

F-2.5 Calculation

The content of hydrocyanic acid in percent shall be found by dropping a perpendicular from the point of intersection of the horizontal line of the measured optical density and the calibration curve.

F-3 METHOD B

F-3.1 Outline of the Method

Hydrogen cyanide is extracted from the sample as a water-soluble salt by means of a solution containing sodium hydroxide, ammonium hydroxide and potassium iodide. The aqueous extract is then titrated with standard silver nitrate until all the cyanide is complexed as the soluble $[Ag(CN)_2]^-$.

The first excess of silver, the end point, is indicated visually with the formation of insoluble silver iodide (turbidity). The hydrogen cyanide content is calculated from the quantity of silver nitrate consumed.

F-3.2 Apparatus

F-3.2.1 *Separatory Funnel* — 250 ml

F-3.2.2. *Burette*, *Microburette* — 10 ml

F-3.2.3 *Erlenmeyer Flasks* — 250 ml

F-3.2.4 *Graduated Cylinder* — 100 ml

F-3.2.5 *Volumetric Flasks* — 1 000 ml

F-3.2.6 *Bottle*, *plastic* — 1 000 ml

F-3.2.7 *Analytical Balance*

F-3.3 Reagents

F-3.3.1 *Silver Nitrate* ($AgNO_3$) — 0.01 N

Dilute 100 ml of 0.1 N silver nitrate (standardized) to 1 litre with water

F-3.3.2 *Potassium Iodide*

F-3.3.3 *Sodium Hydroxide*

F-3.3.4 *Ammonium Hydroxide*

F-3.3.5 *Caustic-Iodide Solution*

Dissolve (3.6 ± 1) g of potassium iodide and 44.1 g of sodium hydroxide in 700 ml of water. Add 180 ml of concentrated ammonium hydroxide and dilute to 1 litre with water. Solutions are stable for at least six months.

F-3.4 Procedure

Take 100 ml of caustic iodide into 250 ml erlenmeyer flask, using the graduated cylinder. Blank the caustic iodide by slowly titrating with 0.01 N silver nitrate to a slight opalescence that persists. Transfer the contents of the erlenmeyer flask to the 250 ml separatory funnel. Add 100 ml of sample with a graduated cylinder. Stopper and shake the separatory funnel for 1 min. Be sure to vent the funnel to relieve pressure. Then let the funnel stand in a holder until the layers separate. Draw off the bottom (aqueous) layer into a 250 ml erlenmeyer flask. Slowly titrate contents of the erlenmeyer flask with standard 0.01 N silver nitrate until an opalescence is obtained. Record this volume of silver nitrate.

NOTE — Avoid breathing acrylonitrile vapors or ammonia vapors from the caustic iodide reagent. Use a well-ventilated hood.

F-3.5 Calculation

Hydrocyanic acid (HCN) content is calculated as:

$$\text{HCN, percent by mass} = \frac{V_1 \text{ x } N \text{ x } 0.054}{V_2 \text{ x } 0.807} \text{ x } 10^2$$

where

V_1 = volume of silver nitrate used in the titration, in ml;

V_2 = volume of sample, in ml;

N = normality of silver nitrate used for titration;

0.054 = milliequivalent weight of hydrogen cyanide (HCN); and

0.807 = density of acrylonitrile, in g/ml.

ANNEX G

[*Table* 1 and *Sl No.* (xv)]

DETERMINATION OF METHYL VINYL KETONE CONTENT

G-1 APPARATUS

G-1.1 Infrared Spectrophotometer — with sodium chloride cell of 0.5 mm path

G-2 REAGENTS

G-2.1 Acrylonitrile — free from methyl vinyl ketone

G-2.2 Methyl Vinyl Ketone

G-3 CALIBRATION CURVE

To plot the calibration curve, prepare solutions of acrylonitrile containing 0.05 percent, 0.1 percent, 0.2 percent, 0.3 percent and 0.4 percent of methyl vinyl ketone. Take the spectrum for the resulting solutions in the range of wavelengths of 7.6 µm to 9.0 µm and determine the optical density of the 8.44 µm analysis line of the solution in the sodium chloride cell. The values obtained shall be plotted against the concentration of methyl vinyl ketone in percent as abscissa and the optical density as ordinate.

G-4 PROCEDURE

Introduce the material under test into the cell. Take the spectrum in the range of wavelength of 7.6 µm to 9.0 µm and determine the optical density of the 8.44 µm analysis line. The amount of methyl vinyl ketone shall be found by dropping a perpendicular from the point of intersection of the horizontal line of the optical density obtained and the calibration curve.

ANNEX H

[*Table* 1 and *Sl No.* (xvi)]

DETERMINATION OF ACETALDEHYDE

H-1 GENERAL

Two methods namely, Method A Determination of acetaldehyde by using schiff reagents, Method B Determination of acetaldehyde by 3-methyl-2-benzothiazolinone hydrazone hydrochloride (MBTH) color reagent and Annex D Gas chromatographic method have been prescribed. In case of disputes, Annex D shall be the referee method.

H-2 METHOD A

H-2.1 Apparatus

H-2.1.1 UV-*Spectrophotometer*

H-2.2 Reagents

H-2.2.1 *Schiff's reagents* — prepared by mixing equal volumes of solution A and solution B as below:

a) Solution A — 0.1 percent solution of fuchsin in water.

b) Solution B — prepared by introducing 6.5 g of sodium pyrosulphate into a 100 ml measuring flask containing 65 ml of a 1 N solution of hydrochloric acid and diluting the resulting solution with distilled water to the mark.

Cool, solution A and solution B to a temperature of 10 ºC, combine them and allow the mixture to stand at a temperature not exceeding 15 °C until it is completely decolorized. Then filter the Schiff's reagent and keep the filtrate at a temperature not exceeding 15 °C in a brown glass bottle.

H-2.2.2 *Acrylonitrile* — free from acetaldehyde

H-2.3 Plotting the Calibration Curve

To plot the calibration curve, prepare solutions of acetaldehyde in acrylonitrile, having the concentrations as 0.05 percent, 0.02 percent, 0.01 percent, 0.005 percent and 0.001 percent.

Pipette 20 ml of each solution in 25 ml measuring flasks, then add 2 ml of schiff's reagent to each flask and dilute each solution to the mark with water. After allowing the solution to stand for 1 h at a temperature of 8 ºC, determine the optical density of each solution at a wavelength of 590 nm, the height of the solution in the cell being 10 mm.

The values obtained shall be plotted with optical density as ordinates and the concentration of acetaldehyde in percent as abscissas.

H-2.4 Procedure

Pipette 20 ml of the sample under test into a 25 ml measuring flask, and 2 ml of schiff's reagent and add water to the mark. After allowing the solution to stand for 1 h at a temperature of 8 °C, measure the optical density at a wavelength of 590 nm, the height of the solution in the cell being 10 mm.

The percentage of acetaldehyde in the sample of acrylonitrile under test shall be found with the aid of the calibration curve from the optical density value obtained.

H-3 METHOD B

H-3.1 General

H-3.1.1 The aldehydes in the sample are reacted with 3-methyl-2-benzothiazolinone hydrazone hydrochloride (MBTH) and then with a ferric chloride, sulfamic acid oxidizer to form a blue color whose intensity is proportional to the concentration of aldehydes present. The intensity of the blue color is measure spectrophotometrically at about 620 nm and is converted to ppm aldehydes as acetaldehyde by means of a calibration curve prepared from standards of known concentrations of acetaldehyde.

H-3.1.2 The inhibitor, 4-methoxyphenol (MEHQ), usually present in acrylonitrile, will react with the oxidizing reagent to produce a colour that interferes with this procedure. The inhibitor is removed by washing the sample with dilute caustic or by distillation. About 65 percent of the acrolein present will also react.

H-3.2 Apparatus

H-3.2.1 *UV-Spectrophotometer*

H-3.2.2 *Pyrex Cells* — 10 mm

H-3.2.3 *Syringes* — 50 ml; 10 ml.

H-3.2.4 *Balance* — analytical sensitive to 0.1 mg

H-3.2.5 *Volumetric Flasks* — 50 ml

H-3.2.6 *Pipettes* — 1 ml, 2 ml and 25 ml.

H-3.3 Reagents

H-3.3.1 *3-Methyl-2-benzothiazolinone Hydrazone Hydrochloride (MBTH) color reagent (0.08 g/l)*

Dissolve 0.08 g of 3-methyl-2-benzothiazolinone hydrazone hydrochloride in water and dilute it up to 1 000 ml. Store in a brown bottle.

H-3.3.2 *Oxidizer Reagent*

Add 8.33 g ferric chloride hexahydrate ($FeCl_3$. $6H_2O$) and 8.0 g sulfamic acid to a 1000 ml volumetric flask. Dissolve in water and dilute it up to volume. Store in a brown bottle.

H-3.3.3 *Acetaldehyde*

This should be re-distilled prior to use as a standard at least every month. This may be done using helium as a carrier gas and an ice water solution as the coolant for the condenser.

H-3.3.4 *4-Methoxyphenol (MEHQ)* — CP grade

H-3.3.5 *3-Methyl-2-benzothiazolinone Hydrazone Hydrochloride*

H-3.3.6 *Ferric Chloride Hexahydrate* — ACS reagent grade

H-3.3.7 *Sulfamic Acid* — 99 percent

H-3.3.8 *Acrylonitrile* — Inhibitor free

H-3.4 Procedure

Pipette 1.0 ml of the sample into 50 ml volumetric flask. Pipette 25 ml of MBTH solution into flask and wait for 45 min to 50 min. Add 2 ml of the oxidizer reagent into flask. Dilute to volume with water and mix thoroughly and wait for 20-21 min. Measure the absorbance of the sample at the maximum a wavelength of 620 nm in the same manner as the standards.

H-3.5 Calculation

Calculate the value from the slope as:

Acetaldehyde content, percent by mass = 1 x
Sample Absorbance Slope

ANNEX J

[*Table* 1 and *Sl No.* (xvii)]

DETERMINATION OF MONOMETHYL ETHER OF HYDROQUINONE (MEHQ)

J-1 GENERAL

Two method namely, Method A determination of monomethyl ether of hydroquinone by spectrophotometer when absorbance is measured at 420 nm and Method B determination of monomethyl ether of hydroquinone by spectrophotometer when absorbance is measured at 295 nm have been prescribed. In case of disputes, method A shall be the referee method.

J-2 METHOD A

J-2.1 Apparatus

J-2.1.1 *UV Spectrophotometer*

J-2.1.2 *Measuring Flasks* — 50 ml

J-2.1.3 *Pipette* — 5 ml

J-2.2 Reagents

J-2.2.1 *Glacial Acetic Acid*

J-2.2.2 *Saturated Sodium Nitrite Solution*

J-2.3 Calibration Curve

To plot the calibration curve, prepare solutions of MEHQ in inhibitor-free acrylonitrile, by accurately weighing (0.10 $\pm$ 0.05) g of MEHQ for stock 1 and (0.40 $\pm$ 0.05) g of MEHQ for stock 2 into separate 100 ml volumetric flasks and then diluting it with inhibitor-free acrylonitrile.

Pipette 1.0 ml, 2.0 ml and 3.0 ml of stock 1 ml and 1.0 ml of stock 2 ml into 100 ml measuring flask and dilute it to volume with acrylonitrile. The standards should be 12 ppm, 25 ppm, 37 ppm and 50 ppm MEQH.

Determine the absorbance of each solution at a wavelength of 420 nm using 10 mm in the cell being 10 mm.

The values obtained shall be plotted with absorbance as abscissa and the concentration of MEHQ in ppm as ordinates.

J-2.4 Procedure

Pipette 5 ml of the sample in 50 ml measuring flask, add 10 ml of glacial acetic acid using a pipette and further add 0.5 ml of saturated sodium nitrite solution. Shake the flask to mix well and allow to stand exactly for 1 min and dilute with water to 50 ml.

After 15 min, measure the extinction on UV-spectrophotometer at 420 nm of the solution above, using 10 mm cell, and compare with that of the blank solution which is prepared by mixing 10 ml of glacial acetic acid, 0.5 ml of saturated sodium nitrite solution and water added to make it a total of 50 ml.

J-2.5 Calculation

Calculate the monomethyl ether of hydroquinone (MEHQ) content by the following formula:

Monomethyl ether of hydroquinone content

$$\text{(MEHQ), ppm} = \frac{M_1}{M_2}$$

where

M_1 = mass of MEHQ determined by the calibration curve, in g; and

M_2 = mass of the sample (5 ml × 0.8), in g.

J-3 METHOD B

J-3.1 Apparatus

J-3.1.1 *UV Spectrophotometer*

J-3.1.2 *Volumetric Flasks* — glass stoppered, 100 ml

J-3.1.3 *Burette*, 25 ml capacity — graduated in 0.1 ml sub-divisions

J-3.1.4 *Absorption Cell* —10 mm path cells

J-3.1.5 *Analytical Balance* — sensitive to ± 0.1 mg

J-3.1.6 *Separatory Funnel* — 500 ml

J-3.2 Reagents

J-3.2.1 *Monomethyl Ether Hydroquinone* (*MEHQ*)

J-3.2.2 *Sodium Hydroxide (1 meq/ml)* — Dissolve 40 g of sodium hydroxide (NaOH) in about 500 ml of water. Cool and then dilute it to 1 litre with water and mix.

J-3.3 Calibration

J-3.3.1 *Preparation of Inhibitor-Free Acrylonitrile*

J-3.3.1.1 Take 200 ml of 1 meq/ml sodium hydroxide and 100 ml acrylonitrile in a 500 ml separatory funnel. Shake the content and leave it to form layers. Once layer gets formed, extract and reserve the acrylonitrile layer. Repeat the extraction till 500 ml of inhibitor-free acrylonitrile is obtained.

J-3.3.1.2 Alternately, distill 750 ml of acrylonitrile and retain 500 ml of the heart-cut.

> CAUTION — For acrylonitrile to be inhibitor free, absorbance at 295 nm shall be less than 0.01. Repeat the process (see **J-3.3.1.1** or **J-3.3.1.2**) if higher than 0.01.

J-3.3.2 *Preparation of Calibration Curve*

J-3.3.2.1 In 100 ml volumetric flask, take 0.160 g of MEHQ and 50 ml inhibitor-free acrylonitrile and stir it to dissolve. Dilute it up to the mark with with inhibitor-free acrylonitrile and mix well. The solution contains 2 000 ppm MEHQ.

J-3.3.2.2 Pipet 10 ml of this solution into another 100 ml volumetric flask. Dilute it up to the mark with inhibitor-free acrylonitrile and mix well. This solution contains 200 ppm MEHQ.

J-3.3.2.3 Using a burette, take 5.0 ml, 7.5 ml, 10.0 ml, and 12.5 ml of the 200 ppm MEHQ solution (**J-3.3.2.2**) into separate 50.0 ml volumetric flasks. Dilute each up to the mark with inhibitor-free acrylonitrile and mix well. These solutions contain 20 ppm, 30 ppm, 40 ppm, and 50 ppm MEHQ respectively.

J-3.3.2.4 Measure the absorbance of each of the standards as prepared above at 295 nm using 1 cm cells and inhibitor-free acrylonitrile as the reference. Prepare a calibration curve by plotting the absorbance of each standard (ordinate) versus the ppm MEHQ (abscissa) and draw the best straight line through the plotted points.

J-3.4 Procedure

Take sample and measure the absorbance at 295 nm using 1 cm cells and inhibitor-free acrylonitrile as a reference.

J-3.5 Calculation

The value of ppm MEHQ present in the sample is determined by reference to the standard calibration curve.

ANNEX K

[*Table* 1 and *Sl No.* (xviii)]

DETERMINATION OF PEROXIDE CONTENT

K-1 APPARATUS

K-1.1 Erlenmeyer Flask, Glass Stoppered — 125 ml capacity

K-1.2 Spectrophotometer — visible with 10 mm path cells

K-2 REAGENTS

K-2.1 Potassium Iodide — reagent grade

K-2.2 Acetic Anhydride

K-2.3 Hydrogen Peroxide — 30 percent chemically pure

K-2.4 Ion-Exchange Resin — any suitable ion-exchange resin may be used

K-3 PROCEDURE

Pipette 25 ml each of sample and peroxide free acrylonitrile in two separate 125 ml glass stoppered Erlenmeyer flasks (peroxide free acrylonitrile can be prepared by passing acrylonitrile through a column of ion-exchange resin). Add 5 ml of acetic anhydride to each flask by pipette. Add 0.5 g of potassium iodide to each flask. Stopper the flask and swirl for 1 min. Fill the two cells with blank and sample and find out absorbance exactly 20 min after addition of potassium iodide. Find out absorbance at 365 nm adjusting zero of the instruments by blank.

K-4 PREPARATION OF CALIBRATION CURVE

Dilute 6.0 ml of 30 percent hydrogen peroxide (H_2O_2) to 100 ml with distilled water (solution A). Dilute 10 ml of solution A to 1 litre by distilled water (solution B). Further dilute 4 ml of solution B to 1 litre with acrylonitrile (solution C). Dilute 10 ml, 30 ml, 50 ml, 70 ml, 90 ml of solution C to 100 ml with acrylonitrile. These solutions are equal to 0.1 ppm, 0.3 ppm, 0.5 ppm, 0.7 ppm and 0.9 ppm of peroxide, respectively. Analyze these standard solutions as given in **K-3** and plot a curve of absorbance against ppm.

K-5 CALCULATION

Find out ppm of peroxide for the sample from the calibration curve.

ANNEX L

[*Table* 1 and *Sl No.* (xix)]

DETERMINATION OF SOLUBLE IRON CONTENT

L-1 APPARATUS

L-1.1 UV-Spectrophotometer

L-2 REAGENTS

L-2.1 Hydrochloric Acid Solution (1 : 1)

L-2.2 Hydroxylamine Hydrochloride Solution — 10 percent

L-2.3 Orthophenanthroline Solution — 0.1 percent

L-2.4 Standard Iron Solution — 1 ml of solution shall contain 0.1 mg of iron

L-2.5 Aqueous Ammonia

L-3 PLOTTING THE CALIBRATION CURVE

Using a burette, introduce 0.5 ml, 1.0 ml, 1.5 ml, 2.0 ml and 3.0 ml of the solution containing iron, respectively, into 100 ml measuring flasks. Then add 9 ml of the hydroxylamine hydrochloride solution to each flask and insert a strip (1 cm wide) of congo red indicator paper in each solution.

Neutralize the solutions with aqueous ammonia until the indicator paper turns red. Further, add 10 ml of orthophenanthroline solution to each flask and dilute the solution with water to the mark. Mix the solution thoroughly.

Determine the optical density of the resulting solution in spectrophotometer at a wavelength 510 nm, the height of the solution being 10 mm. The solution taken for comparison shall be distilled water.

The test results obtained shall be plotted with optical density as ordinates and the content of iron in µg in 5 ml of the solution as abscissas.

L-4 PROCEDURE

Filter the sample under test through a glass funnel with a no. 3 porous plate that is G3 porous plate to free it from impurities. Evaporate 100 ml of the clear filtrate to dryness in a quartz dish on a steam bath. Place the dish in a muffle furnace and keep it there at a temperature of 500 ºC to 550 ºC for 15 min. After cooling, add 1 ml of the hydrochloric acid solution to the dry residue, partly cover the dish with a watch glass and evaporate on a steam bath for 30 min. Then wash the glass with water allowing the wash water to drain into the dish and evaporate until one drop remains. Add 1 ml of the hydroxylamine hydrochloride solution (**L-2.2**) into the dish, insert a strip of the congo red indicator paper and add aqueous ammonia (1 or 2 drops) until the indicator paper turns red. Further, add to the dish, 1 ml each of water and orthophenanthroline solution (**L-2.3**), transfer the solution to a 5 ml measuring flask and dilute the solution with water to the mark and mix. Determine the optical density of the resulting solution in a spectrophotometer at a wavelength 510 nm, the height of the solution being 10 mm. Distilled water shall be taken as the reference solution. By dropping a perpendicular from the point of intersection of the horizontal line of the measured optical density and the calibration curve, find the iron content in micrograms in 5 ml of the solution.

L-5 CALCULATION

The content of soluble iron in percent mass shall be calculated from the formula:

Soluble iron content, percent by mass =

$$\frac{C \times 100}{0.8 \times V \times 1000000}$$

where

C = amount of iron in 5 ml of the solution found from the calibration curve, in µg;

V = volume of acrylonitrile taken for the test, in ml; and

0.8 = density of the acrylonitrile

ANNEX M

[*Table* 1 and *Sl No.* (xx)]

DETERMINATION OF HEAVY METALS (*as* Cu)

M-1 APPARATUS

M-1.1 Spectrophotometer — with 10 mm path cells or equivalent

M-1.2 *p*H Meter

M-2 REAGENTS

M-2.1 Perchloric Acid — 70 percent, reagent grade

M-2.2 Ammonium Citrate Solution — 20 percent

Dissolve 80 g of reagent grade ammonium citrate in 360 ml of distilled water. Filter if solution is cloudy

M-2.3 Sodium Diethyldithiocarbamate — 0.1 percent solution

Dissolve 0.1 g of sodium diethyldithiocarbamate in distilled water and make up to 100 ml

M-2.4 Copper Stock Solution — 5 µg/ml

Dissolve 0.1965 g of reagent grade $CuSO_4.5H_2O$ in distilled water and dilute 100 ml of above solution to 1 000 ml in volumetric flask with distilled water

M-2.5 Acid Mixture

Mix 5 parts concentrated sulphuric acid (H_2SO_4) with 2 parts concentrated nitric acid (HNO_3)

M-3 PROCEDURE

Transfer 125 ml of sample in 250 ml beaker and evaporate on water bath in fume cupboard. Cool the beaker and add 3 ml of acid mixture. Place beaker on hot plate and allow to boil. Cool and further add 0.2 ml of 70 percent perchloric acid ($HClO_4$). Again place on hot plate and heat to fumes. If the residue is coloured after cooling, repeat acid addition. Transfer the contents to 100 ml beaker and make up the volume to 10 ml. Add 5 ml of 20 percent ammonium citrate and adjust the *p*H to 9.1 ± 0.1 with concentrated ammonium hydroxide (NH_4OH). Transfer quantitatively to 25 ml volumetric flask. Add 1 ml of carbamate solution and dilute to mark. Mix well. Record absorbance using 10 mm path cells at 488 nm. Adjust the zero against distilled water blank.

M-4 PREPARATION OF CALIBRATION CURVE

Pipette 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of copper stock solution 5 µg/ml in separate 100 ml beakers. Add 5 ml of ammonium citrate and adjust the *p*H to 9.1 ± 0.1 with concentrated NH_4OH. Transfer the contents of the above beaker to separate 25 ml volumetric flasks and proceed as given in **M-3**. Plot a graph of absorbance against microgram.

M-5 CALCULATION

Read µg of copper for the absorbance of sample solution from the calibration curve.

$$\text{Cu, ppm} = \frac{X}{V \times 0.8}$$

where

X = mass of copper at absorbance of 488 nm, in µg; and
V = volume of sample, in ml.

ANNEX N

[*Table* 1 and *Sl No.* (xxi)]

DETERMINATION OF *p*H (5 PERCENT AQUEOUS SOLUTION)

N-1 APPARATUS

N-1.1 *p*H Meter

N-1.2 Magnetic Stirrer with Glass Stirring Bars

N-1.3 Beaker — 100 ml

N-1.4 Pipette — 5 ml capacity

N-2 REAGENTS

N-2.1 Reference Buffer Solutions — standards *p*H 4 and 7

N-3 PROCEDURE

Transfer 76 ml of distilled water to 100 ml glass breaker. Using 5 ml pipette, transfer 5 ml sample to above beaker. Place the beaker on magnetic stirrer with glass stirring bar. Allow acrylonitrile to dissolve. Immerse the electrode of *p*H meter in the above solution. Avoid the entrainment of any bubbles. Measure *p*H on *p*H meter. Calibrate the *p*H meter with the standard buffer solutions before actual measurement of *p*H of solution.

ANNEX P

[*Table* 1 and *Sl No.* (xxii)]

DETERMINATION OF NON-VOLATILE MATTER

P-1 APPARATUS

P-1.1 Evaporating Dish — 200 ml capacity made of glass or porcelain

P-1.2 Warming Plate — capable of maintaining a temperature of (80 ± 2) °C. Heating element of plate should be enclosed so that acrylonitrile fumes cannot be ignited. The warming plate must be located in fume cupboard.

P-1.3 Constant Temperature Oven — capable of maintaining temperature of (105 ± 2) °C

P-1.4 Desiccator

P-1.5 Pair of Tongs — for handling evaporating dishes

P-1.6 Graduated Cylinder — 100 ml

P-2 REAGENT

P-2.1 Desiccant — indicating type

P-3 PROCEDURE

Clean the evaporating dish and dry at (105 ± 2) °C for 30 min. Remove the dish and cool it in desiccator for 30 min. Weigh the dish up to 0.1 mg accuracy. Measure 125 ml of sample by graduated cylinder and pour it in evaporating dish. Place the dish on warming plate at (80 ± 2) °C. Allow the sample to evaporate in the fume cupboard. Place the dish in oven at (105 ± 2) °C for 30 min. Remove the dish and place in desiccator for 30 min. Weigh the dish up to 0.1 mg accuracy.

P-4 CALCULATION

Calculate the concentration of nonvolatile matter in percent (*m/m*).
Non-volatile matter, percent by mass =

$$\frac{(M_2 - M_1) \times 100}{V \times 0.8}$$

where

M_1 = mass of empty evaporating dish, in g;
M_2 = mass of evaporating dish with nonvolatile matter, in g; and
V = volume of sample, in ml.

ANNEX Q

(*Clause* 3.2.1)

DETERMINATION OF HYDROQUINONE CONTENT

Q-1 OUTLINE OF THE METHOD

This method of test describes a procedure for determining the hydroquinone content in the range of 1 ppm to 20 ppm. In this procedure, the material is evaporated either by air drying at room temperature or vaccum evaporation at 75 °C. The resulting hydroquinone residue is dissolved in water and titrated with dilute standardized ceric acid sulphate solution.

Q-2 APPARATUS

Q-2.1 Rotary Film Evaporator

Q-3 REAGENTS

Q-3.1 Standard Solution of Ceric Acid Sulphate — 0.002 N

Dissolve 1.096 g of ceric ammonium nitrate [$(NH_4)_2Ce(NO_3)_6$] in 20 ml concentrated sulphuric acid in a 50 ml beaker. Slowly pour the ceric solution, while stirring, into 200 ml of water contained in a 600 ml beaker. When the solution is complete, transfer this mixture to a 1 litre volumetric flask and dilute to mix with water.

Q-3.2 Diphenylamine Indicator

Dissolve 0.1 g of diphenylamine in 100 ml concentrated sulphuric acid. Store this solution in a brown bottle.

Q-3.3 Standard Hydroquinone Solution

Dissolve 0.2 g of hydroquinone weighed nearest to 0.1 mg in water and dilute to 1 litre in a volumetric flask. This solution is unstable and should be discarded after one week of normal use.

Q-4 PROCEDURE

Q-4.1 Pipette 50 ml of acrylonitrile into each of the two 125 ml flasks. Connect the flasks to rotofilm evaporator and apply vacuum. Keep the flask in a hot water bath maintained at (75 ± 5) °C. Evaporate the sample to dryness. Do not exceed temperature of 80 °C or 30 min of evaporation time. Carefully repressure the flask and disconnect. Displace any remaining vapours by carefully purging the flask with dry air/nitrogen. Dissolve the residual hydroquinone in 25 ml water. Add 3 drops of diphenylamine indicator using the same dropper every time. Titrate each solution with 0.002 N ceric acid sulphate to a faint blue point which persists for 15 s.

Pipette 10 ml of standard hydroquinone solution (**Q-3.3**) into each of two 125 ml Erlenmeyer flasks. Add 3 drops of diphenylamine indicator using the same dropper as used for the test and titrate with 0.002 N ceric acid sulphate solution to a faint blue point which persists for 15 s.

NOTE — Duplicate titrations should agree within 0.05 ml. Use the average of the two values.

Q-5 CALCULATION

Hydroquinone content in ppm = $\frac{V \times F}{M} \times 100$

where

V = volume of ceric acid sulphate solution used to titrate the sample, in ml;

F = Factor = $\frac{mg\ of\ hydroquinone\ in\ 10\ ml\ of\ sample}{volume\ ceric\ acid\ sulphate\ solution\ required\ to\ titrate\ the\ standard\ solution\ (ml)}$; and

M = mass of sample taken, in g.

Q-6 REPORT

Report the hydroquinone content nearest to 0.1 ppm.

ANNEX R

(*Clause* 6.1)

SAMPLING OF ACRYLONITRILE, TECHNICAL

R-1 GENERAL REQUIREMENTS OF SAMPLING

R-1.1 In addition to precautions given in **4** while preparing, storing and handling samples, the following precautions and directions shall be observed.

R-1.2 The persons sampling and testing the material shall wear suitable working clothes, protective eye glasses, rubber gloves and gas mask.

R-1.3 Sampling shall be carried out with the aid of special sampling instrument or clean glass tubes, taking equal subsamples from the lower, middle and upper layers of the product.

R-l.4 The samples shall be placed in clean and dry bottles provided with a ground-glass stopper.

R-1.5 Each sample container shall be sealed airtight after tilling and marked with full identification particulars, such as, the name of the product, the sample number, the date of sampling, lot number, date of manufacture of the materials, etc.

R-2 SCALE OF SAMPLING

R-2.1 Filling of the Material in Tanks

A representative sample shall be taken from each tank. The total volume of the sample taken from each tank shall not be less than 1.5 litres. The sample shall be thoroughly mixed and divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

R-2.1.1 The material in a tank shall be considered as conforming to the requirements of the specification if the test sample meets all the requirements given in the specification.

R-2.2 Packing of Material in Steel Cans and Drums

R-2.2.1 *Lot*

In a single consignment, all the containers belonging to the same batch of manufacture shall constitute a lot. If the consignment consists of different batches of manufacture, the containers belonging to the same batch of manufacture shall be separated and shall constitute separate lots.

R-2.2.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately.

R-2.2.3 The number of containers to be selected (n) for this purpose shall depend on the size of the lot (N) and shall be in accordance with Table 2.

R-2.2.3.1 The containers shall be selected from the lot at random. In order to ensure the randomness of selection, a reference may be made to IS 4905. In case this standard is not readily available, following procedure may be adopted:

Starting from any container in the lot, count them as 1,2,3 . . . . .and so on up to r in one order where r is the integral part of N/n, every r^{th} container thus counted shall be withdrawn till the requisite number of containers is obtained.

R-2.2.4 *Test Sample and Referee Sample*

R-2.2.4.1 From each of the containers selected according to **R-2.2.3**, approximately equal quantity of material shall be taken and thoroughly mixed so as to form a composite sample. The total volume of the composite sample shall be not less than 1.5 litres. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee. While preparing the sample and transferring it to the sample containers, general precautions given in **R-1** shall be observed.

R-2.2.4.2 *Referee Sample*

The composite sample marked for this purpose shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the two so as to be used in case of a dispute.

R-2.2.5 *Number of Tests*

Test for determining all the requirements given in **3** shall be carried out on the composite sample.

R-2.2.6 *Criteria for Conformity*

A lot shall be declared as conforming to the requirements of the specification, if all the test results on the composite sample meet the corresponding requirements given in Table 1.

Table 2 Scale of Sampling for Containers
(*Clause* R-2.2.3)

Sl No. (1)	**Lot Size** *N* (2)	**No. of Containers to be Selected** *n* (3)
i)	up to 25	3
ii)	26 to 50	5
iii)	51 to100	8
iv)	101 to 300	13
v)	301 to 500	20
vi)	501 and above	32

ANNEX S

(*Foreword*)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative*(*s*)
National Chemical Laboratory (NCL), Pune	DR C. V. RODE (***Chairperson***)
Alkyl Amines Chemicals Ltd, Mumbai	SHRI S. V. NIKUMBHE SHRI SAMEER KATDARE (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	SHRI K. L. RAPHAEL SHRI KIRTI GAJJAR (*Alternate*)
All India Distillers Association (AIDA), New Delhi	SHRI V. N. RAINA
BASF India Limited, Mumbai	SHRI KIRAN BHAT SHRI HEMAL (*Alternate*)
CSIR-Central Drug Research Institute (CDRI), Lucknow	DR SANJEEV KANOJIYA
Chemical And Petrochemicals Manufacturers Association (CPMA), New Delhi	SHRI UDAY CHAND
Deepak Fertilizer, New Delhi	DR L. B. YADAWA SHRI SURESH AMLE (*Alternate*)
Deepak Phenolics Ltd, Vadodara	SHRI DHARMESH SIDDHAPURIA SHRI SANDIP KUMAR PANDYA (*Alternate*)
Dow Chemical International Pvt Ltd, Mumbai	SHRI V. MOHANDOSS SHRI GOVIND GUPTA (*Alternate*)
Godavari Biorefineries, Mumbai	SHRI SHANUL LAXMANRAO PAGAR SHRI APPASAHEB J. WANI (*Alternate*)
Gujarat Narmada Valley Fertilizers Company Ltd, Ahmedabad	DR R. M. PATEL SHRI C. S. PATEL (*Alternate*)
Hindustan Organic Chemicals Ltd (HOCL), Mumbai	SHRI DELEEP KUMAR. K. SHRI PRADEESH NARAYANAN (*Alternate*)
INEOS Styrolution India Ltd, Vadodara	DR KANAK DASS
India Glycols Ltd, Uttarakhand	DR R. K. SHARMA SHRI ALOK SINGHAL (*Alternate*)
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